

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

In Re Application of :
Masayoshi HANNA et al. : Confirmation No.: 008270
Serial No.: 10/501,507 : Group Art Unit: 1713
Filed: July 15, 2004 : Examiner: BERNSHTEYN,
Michael

For: PROCESS FOR PRODUCING WATER-ABSORBING RESIN

DECLARATION UNDER 37 C.F.R. 1.132

MS RCE
COMMISSIONER FOR PATENTS
P. O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Masayoshi Handa, residing in Hyogo-ken, Japan, hereby declares and states as follows:

1. That I am one of the co-inventors of U.S. Application Serial No. 10/501,507 filed on July 15, 2004, entitled PROCESS FOR PRODUCING WATER-ABSORBING RESIN. I am thoroughly familiar with the contents of said Application, its prosecution before the United States Patent and Trademark Office and the references cited therein.

2. That I received a master's degree from Kanazawa University of Japan, faculty of natural science and technology in the year 1994, majoring in material chemistry.

3. That I have been employed in Sumitomo Seika Chemicals Co., Ltd. in the year 1994 and have been assigned to the Research Laboratories.

4. That I have been involved in the research and development of water-absorbent resin since 1998.

5. That the following experiments were conducted by myself or under my direct supervision and control in order to verify that there is a remarkable effect in adding a reducing agent after the polymerization as compared to a conventional technique of adding a reducing agent before the polymerization as taught by Tsuchiya et al. (U.S. Patent Application Publication No.

US 2002/0034911 A1.

EXPERIMENTAL METHOD

The following comparative test was conducted.

In the experiment, the same procedures as in Example 3 of the present specification were carried out except that a reducing agent was added before the polymerization.

Specifically, 500 ml of n-heptane was added to a 1000-ml five-necked cylindrical round bottomed flask equipped with a stirrer, a reflux condenser, a dropping funnel, a thermometer and a nitrogen gas inlet tube. Thereto was added 0.92 g of sucrose fatty acid ester (surfactant: S-370, manufactured by MITSUBISHI CHEMICAL CORPORATION) having an HLB of 3.0 and dispersed. The temperature of the dispersion was raised to dissolve the surfactant, and thereafter cooled to 55°C.

Separately from the above, 92 g of a 80% by weight aqueous solution of acrylic acid was added to a 500-ml Erlenmeyer flask. Thereto was added

dropwise 102.2 g of a 30% by weight aqueous sodium hydroxide solution while externally cooling, to neutralize 75% by mol of acrylic acid, to give a partially neutralized salt of acrylic acid. Further, 50.2 g of water, 0.11 g of a polymerization initiator potassium persulfate, and 9.2 mg of a crosslinking agent ethylene glycol diglycidyl ether, and 0.046 g of anhydrous sodium sulfite were added thereto, to give an aqueous solution of a monomer for a first step polymerization.

The entire amount of this aqueous solution of the monomer for a first step polymerization was added to the above-mentioned five-necked cylindrical round bottomed flask under stirring and dispersed. After the internal of the system was sufficiently replaced with nitrogen, the temperature of the mixture was raised, and the polymerization reaction was carried out for 1 hour while keeping its bath temperature at 70°C. Thereafter, the polymerization slurry was cooled to room temperature.

Further, 119.1 g of a 80% by weight aqueous solution of acrylic acid was added to a separate 500-ml Erlenmeyer flask. Thereto was added dropwise 132.2 g of a 30% by weight aqueous sodium hydroxide solution while cooling, to neutralize 75% by mol of acrylic acid. Further, 27.4 g of water, 0.14 g of potassium persulfate, and 35.7 mg of ethylene glycol diglycidyl ether, and 0.060 g of anhydrous sodium sulfite were added thereto, to give an aqueous solution of a monomer for a second step polymerization. The aqueous solution was cooled in an ice water bath.

The entire amount of this aqueous solution of the monomer for a second step polymerization was added to the above-mentioned polymerization slurry. After the internal of the system was again sufficiently replaced with nitrogen, the

temperature of the mixture was raised, and the second-step polymerization reaction was carried out for 2 hours while keeping its bath temperature at 70°C. After the termination of the polymerization, 0.264 g of a 40% by weight aqueous solution of pentasodium diethylenetriaminepentaacetate was added to a water-containing gelated product dispersed in n-heptane under stirring. Subsequently, water of the water-containing gelated product was removed to the external of the system by azeotropic dehydration. To the resulting gelated product was added 8.44 g of a 2% by weight aqueous solution of ethylene glycol diglycidyl ether, and water and n-heptane were further removed from the mixture by distillation, and the residue was dried, to give 213.9 g of a water-absorbent resin.

The yellow index was measured according to the method described in the present specification for each of the resulting water-absorbent resins.

RESULTS AND DISCUSSION

The results are shown hereinbelow.

Also, the data on Example 3 of the present specification are also shown in the table.

	Reducing Agent	Metal Chelating Agent	Yellow Index		
			Immediately After Drying	50°C, 90% RH	After 20 Days
Example 3	Added After Polymerization 0.0625%	Added After Polymerization 0.0625%	7.4		8.2
Comparative Experiment	Added Before Polymerization 0.0625%	Added After Polymerization 0.0625%	9.9		14.1

As is clear from the above results, the action of the reducing agent is significantly different between a case where the reducing agent is added after the polymerization and a case where the reducing agent is added before the polymerization as indicated by the yellow index. The reasons therefor are presumably as follows. In general, when a reducing agent such as a sulfite is added before the polymerization, a redox initiator system is provided with initiator peroxide, thereby accelerating the cleavage of the radical. Since the reducing agent is consumed during the polymerization, it is considered that the reducing agent remains in a very small amount after the polymerization. Therefore, in a reaction system where the oxidizing agent and reducing agent are added before the polymerization as in the case of Tsuchiya, the oxidizing agent and the reducing agent do not remain in effective amounts after the polymerization. Therefore, even if a metal chelating agent is present in such a system, sufficient effects cannot be exhibited.

On the other hand, in the method of adding the reducing agent after the polymerization as taught in the present invention, since effective actions of the oxidizing agent and the reducing agent can take place in the presence of a metal chelating agent, there are some unexpected effects that (1) yellow index shows improvement immediately after drying, and that (2) discoloration resistance is exhibited with the passage of time. Therefore, the present invention exhibits unexpected effects from Tsuchiya.

6. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

7. Further declarant saith not.

Masayoshi Handa
Masayoshi Handa

December 28, 2006
Date